

CLAIMS

What is claimed is:

1. A powder comprising tungsten carbide metal grains, the powder containing:
 - 5 (a) carbon at less than about 6.09 weight percent; and
 - (b) cobalt at less than about 0.6 weight percent; and
 - (c) a grain growth inhibitor selected from a group consisting of vanadium carbide, chromium carbide, titanium carbide, tantalum carbide, molybdenum carbide, or combinations thereof.
- 10 2. The powder of claim 1, wherein said grain growth inhibitor of step (c) is vanadium carbide.
- 15 3. The powder of claim 1, wherein the average diameter of metal grains is less than about 5 micrometers.
4. The powder of claim 1, wherein the average diameter of metal grains is between about 0.1 micrometers and about 0.75 micrometers.
- 20 5. A method of producing a tungsten carbide powder comprising the following steps:
 - (a) forming a metal powder mixture by mixing tungsten particles with carbon powder;
 - (b) ball-milling said metal mixture;
 - (c) adding to said mixture a grain growth inhibitor selected from a group consisting of vanadium carbide, chromium carbide, titanium carbide, tantalum carbide, molybdenum carbide, or combinations thereof;
 - 25 (d) transferring said milled metal powder to a refractory crucible; and

(e) carburizing said milled metal powder to form tungsten carbide particles.

6. The method of claim 5, wherein said tungsten particles of step (a) are selected from a group consisting of metatungstate, peratungstate, and tungstic acid.

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7. The method of claim 5, wherein the metal mixture of step (a) comprises 94 parts by percent tungsten and 6 parts by percent carbon.

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8. The method of claim 5, wherein said milling of step (b) is conducted for at least about 20 hours.

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9. The method of claim 5, wherein said grain growth inhibitor of step (b) is vanadium carbide.

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10. The method of claim 5, wherein said grain growth inhibitor of step (c) is added while conducting the milling of step (b).

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11. The method of claim 5, wherein the step (e) consists of heating said mixture between 1475 degrees Centigrade and 1700 degrees Centigrade in an atmosphere suitable to yield formation of tungsten carbide particles.

12. A method of forming a densified tungsten carbide-containing ceramic body, the method comprising:

(a) milling a tungsten carbide powder comprising tungsten carbide particles in a suitable organic solvent for more than 71 hours, such that rough edges on particles in the powder are reduced;

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- (b) adding cobalt to milling mixture at between about 5 percent and about 10 percent into the total milling time to swedge cobalt onto the particles in the powder;
- (c) contacting said granulated powder with a mixture comprising a paraffin-type wax and an ester-type wax;
- (d) spray-drying said milled powder under conditions sufficient to granulate the tungsten carbide particles into a spherical shape;
- (e) compacting said granulated tungsten carbide powder under conditions sufficient to form a preform ceramic body;
- 10 (f) removing said paraffin-type wax and ester-type wax from said preform ceramic body, thereby forming a debindered population of tungsten carbide particles; and
- (g) sintering said debindered preform ceramic body, thereby forming a densified tungsten carbide-containing ceramic body.

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13. The method of claim 12, wherein said tungsten carbide powder of step (a) is ball-milled and spray-dried repeatedly for between about 100 hours and about 400 hours.
14. The method of claim 12, wherein said tungsten carbide powder of step (a) is ball-milled and spray dried repeatedly for between about 150 hours and about 350 hours.

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15. The method of claim 12, wherein the tungsten carbide powder according to step (a) is the powder of claim 1.
16. The method of claim 12, wherein said organic solvent of step (a) is n-heptane.

17. The method of claim 12, wherein the particle size of the tungsten carbide powder prior to debinding is between about 0.001 micrometers and about 0.6 micrometers.

5 18. The method of claim 12, wherein the ester-type wax is selected from a group consisting of beeswax, montan wax, carnauba wax, and spermaceti.

19. The method of claim 12, wherein the paraffin-type wax is selected from a group consisting of petroleum wax, microcrystalline wax, and hydrocarbon wax.

10 20. The method of claim 18, wherein the ester-type wax is added to less than about 5 weight percent tungsten carbide powder.

15 21. The method of claim 20, wherein the paraffin-type wax is added to greater than about 0.1 weight percent tungsten carbide powder.

20 22. The method of claim 12, wherein debinding of the preform ceramic body is performed *in situ* according to the following steps:

(a) by heating the preform ceramic body, under a pressure of inert gas at a rate of at least about 3.5 degrees Centigrade-per-minute to at least about 280 degrees Centigrade;

(b) holding this temperature for at least about 45 minutes;

(c) increasing the temperature at a rate of at least about 1.5 degrees Centigrade-per-minute to at least about 380 degrees Centigrade;

(d) holding at this temperature for at least about 45 minutes;

(e) increasing the temperature at a rate of at least about 6 degrees Centigrade-per-minute to at least about 480 degrees Centigrade; and

(f) maintaining this temperature for at least about 3 minutes.

23. The method of claim 12, wherein the inert gas is selected from a group consisting of nitrogen, argon, helium, neon, krypton, and xenon.

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24. The method of claim 12, wherein sintering of the preform ceramic body is performed under a vacuum.

10 25. The method of claim 24, wherein the temperature is increased at a rate of at least about 6 degrees Centigrade-per-minute to at least about 1580 degrees Centigrade and maintained at this temperature for at least about 45 minutes, and wherein at least about 28 KSI inert gas pressure is applied and held for at least about 45 minutes.

15 26. The method of claim 25, wherein the inert gas is selected from a group consisting of nitrogen, argon, helium, neon, krypton, and xenon.

27. The method of claim 12, wherein debinding and sintering is performed in a single cycle.

28. A ceramic body prepared by the method of claim 12.

20 29. A ceramic body having a transverse rupture strength of at least about 300,000 pounds-per-square-inch and a Rockwell A-scale hardness of up to about 96 at 20 degrees Centigrade.

30. A ceramic body having a transverse rupture strength of at least about 300,000 pounds-per-square-inch and a Rockwell A-scale hardness of between about 90 and about 96 at 20 degrees Centigrade.

31. A ceramic body having a transverse rupture strength of at least about 300,000 pounds-per-square-inch and a Rockwell A-scale hardness of between about 94 and about 96 at 20 degrees Centigrade.

5 32. A ceramic body having a transverse rupture strength of at least about 300,000 pounds-per-square-inch and a Rockwell A-scale hardness between about 95 and about 96 at 20 degrees Centigrade.

10 33. The ceramic body of claim 28, 29, 30, 31 or 32, wherein the Rockwell A-scale hardness is at least about 90 at 800 degrees Centigrade.

34. The ceramic body of claim 28, 29, 30, 31 or 32, wherein the Rockwell A-scale hardness is at least about 91 at 800 degrees Centigrade.

15 35. The ceramic body of claim 28, 29, 30, 31 or 32, wherein the Rockwell A-scale hardness is at least about 92 at 800 degrees Centigrade.

36. The ceramic body of claim 28, 29, 30, 31 or 32, wherein cobalt is present from about 0.01 weight percent to about 1.0 weight percent.

15 37. The ceramic body of claim 28, 29, 30, 31 or 32, wherein the cobalt content is less than about 0.6 weight percent.

38. The ceramic body of claim 28, 29, 30, 31 or 32, wherein the average tungsten carbide particle size is less than about 1.0 micrometers.

39. The ceramic body of claim 28, 29, 30, 31 or 32, wherein the average tungsten carbide particle size is between about 0.001 micrometers and about 0.6 micrometers.

40. The ceramic body of claim 28, 29, 30, 31 or 32, wherein the ceramic body has a tungsten-bound carbon content of at least about 5.5 weight percent.

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41. The ceramic body of claim 28, 29, 30, 31 or 32, wherein the ceramic body has a density of at least about 95 weight percent of its theoretical density.

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42. The ceramic body of claim 28, 29, 30, 31 or 32, wherein the ceramic body comprises at least about 98 weight percent tungsten carbide.

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43. The ceramic body of claim 28, 29, 30, 31 or 32, wherein the ceramic body comprises at least about 99 weight percent tungsten carbide.

44. The ceramic body of claim 28, 29, 30, 31 or 32, wherein the ceramic body is shaped as a cutting tool.

45. The ceramic body of claim 28, 29, 30, 31 or 32, wherein the ceramic body is shaped as a router bit, knife, or insert.

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46. A method of cutting, machining, or wearing using the ceramic body of claim 28, 29, 30, 31 or 32.

47. The method of claim 46, wherein cutting, machining, or wearing is performed at high-speed.

48. The method of claim 46, wherein the ceramic body is contacted with wood or metal.

49. A preform ceramic body formed according to step (e) of claim 12, comprising tungsten carbide and a mixture of paraffin-type and ester-type waxes.

5 50. The preform ceramic body of claim 49, wherein the ester-type wax is selected from a group consisting of beeswax, montan wax, carnauba wax, and spermaceti.

10 51. The preform ceramic body of claim 49, wherein the paraffin-type wax is selected from a group consisting of petroleum wax, microcrystalline wax, and hydrocarbon wax.

52. The preform ceramic body of claim 49, wherein the ester-type wax comprises less than about 5 weight percent tungsten carbide powder.

15 53. The preform ceramic body of claim 49, wherein the paraffin-type wax comprises greater than about 0.1 weight percent tungsten carbide powder.